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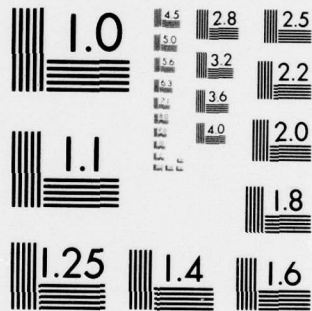
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Effects of the Gaseous Environment on Propagation of
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by

M. M. Nicholson and F. A. Pizzarello

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EFFECTS OF THE GASEOUS ENVIRONMENT ON PROPAGATION OF
ANODIC REACTION BOUNDARIES IN LUTETIUM DIPHTHALOCYANINE FILMS

M. M. Nicholson* and F. A. Pizzarello

Electronics Research Center, Rockwell International, Anaheim, California 92803

ABSTRACT

Influences of the gaseous environment on the anodic oxidation of lutetium diphthalocyanine films contacted by aqueous Na_2SO_4 or KCl were investigated with a moving-boundary technique. Ambient water vapor was required to propagate the red/green boundary from both electrolytes. Oxygen was also required with the sulfate solution but not with the chloride. The results suggest that the diphthalocyanine oxidation product containing sulfate may bind oxygen reversibly at room temperature.

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Key Words: Diphthalocyanines, moving boundary, oxygen, electrochromism

Recent papers from this laboratory have described color-boundary propagation in lutetium diphthalocyanine films due to anodic oxidation of the dye (1,2). The film was supported on a strip of alumina, with the lower end immersed in an aqueous electrolyte and the upper end contacted by gold. Under constant applied current, the color conversion from green to red began at the dye/electrolyte interface and moved upward through the film. The electrochemical reaction was shown, approximately, to be a two-electron oxidation of the diphthalocyanine. Charge compensation occurred by migration of anions from the electrolyte through the red phase under the influence of the electric field. An ionic mobility of $4 \times 10^{-6} \text{ cm}^2/\text{V-sec}$ was determined in the solid oxidation products formed with both chloride and sulfate electrolytes. In radiotracer experiments with Cl^{36} and S^{35} , the chloride product gradually lost chlorine and reverted to green in ambient air, but the sulfate product retained its red color and its radioactivity more than a month (2). It was also found that dry and moist elemental chlorine reacted immediately with lutetium diphthalocyanine films to form a red product (3).

In this paper, we report pronounced dependences of the faradaic boundary propagation on the composition of the gas in contact with the dye film. Specifically, the effects of water vapor and oxygen on the velocity of propagation from chloride and sulfate electrolytes were determined. The results are examined in relation to crystal structures of related actinide diphthalocyanines and the known abilities of certain phthalocyanines and porphyrins to form oxygen adducts. The interesting possibility of oxygen-carrier activity in diphthalocyanine derivatives is noted.

Experimental

Film specimens of lutetium diphthalocyanine [$\text{LuH}(\text{Pc})_2$] were prepared by vacuum sublimation of the dye onto a single-crystal sapphire strips 1.25 cm wide and approximately 5 cm long (1,2). The film thickness was estimated from its optical density at 670 nm in the initial green state.

Quantitative measurements of the boundary position as a function of time were made with the cell shown in Fig. 1. The container was a Klett colorimeter cell, open at the top, with an inlet for blanketing the film with the selected gas. The electrolyte was 1 M Na_2SO_4 or 1 M KCl . In the sulfate solution, the reference and counter electrodes were Pb/PbSO_4 ; in the chloride, they were Ag/AgCl .

Details of the cell-assembly and measurement procedures were described previously (1). Constant anodic currents were applied with a Princeton Applied Research (PAR) 173 galvanostat. The total voltage between the working and reference electrodes was monitored with a Keithly 610C electrometer and strip-chart recorder. With total applied currents in the micro-ampere range, the current density through the cross section of the film ranged from approximately 20 to 50 mA/cm^2 . An open cell, illustrated in Fig. 2, was used to determine effects of the gases impinging directly on the red film. The boundary was then characterized as moving or stationary by observing it under a low-power microscope, and the corresponding instantaneous voltages were noted.

The gases included in the study were ambient air, dry and wet oxygen, and dry and wet helium. In addition to cells with flowing helium, some of the partially enclosed cells were set up in a helium-atmosphere glove box where the oxygen content did not exceed a few ppm.

Results and Discussion

Qualitative observations of the boundary movement and voltage changes are summarized in Table 1. The results were consistent through many experiments performed with a number of cells. Striking ambient effects occurred under certain conditions. For example, dry helium introduced above the sodium sulfate solution could drive the galvanostat to its full compliance of approximately 95 V in a few seconds. Quantitative data for the two electrolytes in partially enclosed cells are presented in Figs. 3 and 4, where the propagation distances and voltages are plotted as functions of time.

It is apparent from the table that some water in the ambient gas was essential for propagation of the color change from both chloride and sulfate electrolytes; dry helium or dry oxygen impinging directly on the red film stopped the boundary movement in each case and caused a major increase in electrical resistance. Moreover, the water content of the film above the liquid could not be attributed to capillary action. The meniscus against the dye was nonwetting, and the previous tracer study had shown the uptake of anions to be stoichiometric (2). It is possible that water also combines with the dye stoichiometrically or that it accompanies the anion in some definite proportion.

The responses of the film to oxygen were quite different with the two electrolytes. Figure 3 represents the behavior in sodium sulfate. The boundary propagation, which had begun in air at point A, stopped completely on introduction of dry helium at B, and the voltage rose

essentially to the compliance of the instrument. On addition of air at C, the voltage dropped abruptly, and normal propagation continued. Wet helium, introduced at D, also arrested the boundary propagation, but the voltage then gradually fell instead of increasing as it did with dry helium. Again, the propagation resumed in the usual voltage range on addition of air at E. This figure establishes the important point that oxygen is necessary for propagation of the boundary from a sulfate solution. Although dry helium stopped all anodic processes, wet helium apparently transferred enough water to the outer surface of the film to support an electrolytic side reaction at low voltage. Oxidation of the dye was then effectively shunted by the lower faradaic resistance.

The contrasting results obtained with potassium chloride are illustrated by Fig. 4. Dry helium, introduced at point B, caused a gradual rise in voltage, but the boundary propagation still continued at the rate observed in ambient air (A-B and C-D). Wet helium, beginning at D, caused a sharp voltage drop and a pronounced decrease in the boundary velocity. This was attributed to electrolytic shunting by a film of water, as in the sodium sulfate experiment. The film in Fig. 4 probably remained somewhat wet, even after the final addition of air at point E.

In summary, an oxygen-containing atmosphere was required to propagate the boundary from sulfate solution, but not from chloride. Dry helium increased the resistance of the film system, and very dry oxygen or very dry helium could stop the boundary propagation from either electrolyte.

These points are borne out by observations of the initial reaction site under different conditions. Figure 1 shows a scribe line in the dye,

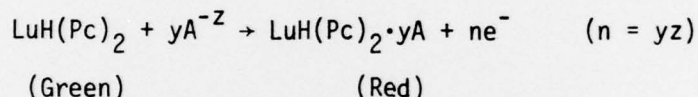
which was placed just below the meniscus to limit any downward propagation of the red color. In sodium sulfate solution, the dye reaction always began at the meniscus. It then travelled upward with oxygen present, but with oxygen absent, it proceeded downward only and stopped at the scribe line. The dye reaction below the liquid level probably was sustained by anodically formed oxygen. Any oxygen escaping into the gas phase was highly diluted, however, so that upward propagation was prevented. In the chloride electrolyte, the red color first appeared within the small submerged area and then continued upward, with or without oxygen present.

The effects of the gas environment were found to be associated almost entirely with the red region, rather than the green. This was apparent from the anion specificity in the case of oxygen. It was indicated in the case of water by Fig. 5, which shows a current-voltage plot determined on a strip of green dye film between two gold contacts. The relationship was ohmic in air up to 93 V (~ 20 V/cm). Wet and dry helium, passed over the film for more than an hour, produced significant resistance changes, but none approaching the magnitude of those in the propagation experiments. It should be noted in this context that other investigators have reported very large ambient effects of water and oxygen on electronic conduction in divalent-metal phthalocyanines (4). However, such compounds generally have resistivities several orders of magnitude higher than those of the rare-earth diphthalocyanines (5) and hence may be more susceptible to ambient effects.

We shall now consider the anodic reactions of the dye, some requirements for migration of anions into the film, and the possibility that the

oxidation product containing sulfate could be an oxygen carrier at room temperature.

Moskalev and Kirin originally suggested that the electrochromism of LuH(Pc)_2 at anodic potentials in KCl was due to complexing with oxygen or water (6). It is now clear that both of these substances are involved with a sulfate electrolyte and that water, but not oxygen, is needed for the anodic process in chloride solution. In addition to any direct addition reactions, charge compensation must accompany the loss of electrons by the dye. Our previous work showed that this was accomplished by migration of anions A^{-z} into the film (1,2):



The experimental n values were approximately 2 for KCl and Na_2SO_4 . Further work will be required to determine whether water and oxygen also participate stoichiometrically in the electrode process and how they are bonded within the organic structure.

The mobility of anions within the red phase may be examined in relation to the space available in the organic solid. Crystal structures of the lanthanide compounds LnH(Pc)_2 apparently have not been determined, but those of U(Pc)_2 and Th(Pc)_2 are known in detail (7,8). Since the lanthanide (III) and actinide (IV) cations are similar in size, their diphthalocyanines probably have similar crystal structures and spacings. The unit cell of U(Pc)_2 has very large dimensions: 18.74, 18.73, and 15.61 Å (7). This crystal contains open spaces great enough to accommodate unsolvated

chloride ions, with a radius of 1.8 \AA . Hence it is plausible that oxidation of LuH(Pc)_2 in a chloride electrolyte could occur, and be reversed, without a major change in lattice dimensions. This is in keeping with the high solid-state chloride-ion mobility of $4 \times 10^{-6} \text{ cm}^2/\text{V-sec}$.

The similar mobility of larger, polyatomic anions such as sulfate in the red phase seems to require further explanation. An attractive possibility, consistent with the ambient-atmosphere dependence, is the formation of an oxygen adduct by $\text{LuH(Pc)}_2 \cdot \text{SO}_4$. It has long been known that certain organic crystals can expand on addition of oxygen. For example, Calvin *et al.* observed pronounced dimensional changes along one crystallographic axis in cobalt^{II} bis-(salicylaldehyde)ethylenediamine during cyclical absorption and desorption of oxygen (9). Such a lattice expansion of lutetium diphthalocyanine might provide the additional space needed for the sulfate ions. This mechanism requires the presence of oxygen throughout the red film--not just at the red/green interface. The very large voltage change that occurred on introduction of helium also suggests an effect in the bulk of the red film.

Thus it is quite possible, but not proved, that the sulfate oxidation product of LuH(Pc)_2 is an oxygen carrier, *i.e.*, a compound that binds oxygen reversibly, at room temperature. Although much work has been done in recent years on synthetic and natural oxygen carriers (10-14), this could be the first instance of carrier activity controllable by anions, or (one may predict) by anion exchange.

Comparisons with known dioxygen complexes of porphyrins and other phthalocyanines are of interest. The natural hemoproteins achieve re-

versible oxygen binding by virtue of complicated structures attached to the porphyrin ring. These features have been simulated in synthetic "picket fence porphyrins" of iron(II) (11). Among simpler molecules, manganese(II) tetraphenylporphyrin forms a reversible dioxygen complex near -79°C in the presence of pyridine (12), but this is unstable at room temperature. The formal description $\text{Mn}^{\text{IV}}(\text{TPP})\text{O}_2^{2-}$ was suggested for the adduct, with $\text{Mn}(\text{TPP})(\text{Py})$ as the pyridine-containing intermediate. Manganese(II) phthalocyanine binds oxygen in pyridine at much higher temperatures. The final product $(\text{Mn}^{\text{III}}\text{PcPy})_2\text{O}$ (13,14) is formed through the intermediate $\text{MnPc}(\text{O}_2)$, which may contain end-bound oxygen (14). This intermediate also can be formed in N,N-dimethylacetamide, where it is very stable unless a base such as dimethylamine is present (14). It then loses oxygen rapidly. These examples bear out the general principle that oxygen-carrier behavior in the porphyrins and phthalocyanines results from a special balance of conditions that is not easily achieved. Our results suggest that this balance may exist in a product such as $\text{LuH}(\text{Pc})_2\cdot\text{SO}_4$ or in a solid hydrate of that product. Further investigations of the diphtalocyanine-oxygen systems are in progress.

Acknowledgment

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ILLUSTRATIONS

1. Partially Enclosed Cell
2. Open Cell .
3. Ambient Effects on Boundary Propagation from 1 M Na_2SO_4
4. Ambient Effects on Boundary Propagation from 1 M KCl
5. Current-Voltage Characteristics of Green Film Between Two Gold Contacts

Table 1. Summary of Gaseous Environment Effects on Boundary Propagation^a

Electrolyte	1 M Na ₂ SO ₄		1 M KCl	
	Boundary Movement	Voltage Change ^b	Boundary Movement	Voltage Change ^b
Air	Yes	--	Yes	--
Dry Helium				
Open ^c	No	Increase	No	Increase
Enclosed ^d	No	Increase	Yes	Slow Increase
Glove Box (Enclosed)	No	--	Yes	--
Wet Helium				
Open	No	None	Yes	None
Enclosed	No	Slow Decrease	Yes (Slow)	Decrease
Dry Oxygen				
Open	No	Increase	No	Increase
Enclosed	Yes	Slow Increase	Yes	Increase
Wet Oxygen				
Open	Yes	Slight Increase	Yes	None
Enclosed	Yes	None	--	--

^aCurrent 0.2 μ A; optical density of green film 0.5 to 0.9 at 670 nm.

^bInitial change relative to voltage in ambient air.

^cOpen cell as in Fig. 2.

^dPartially enclosed cell as in Fig. 1.

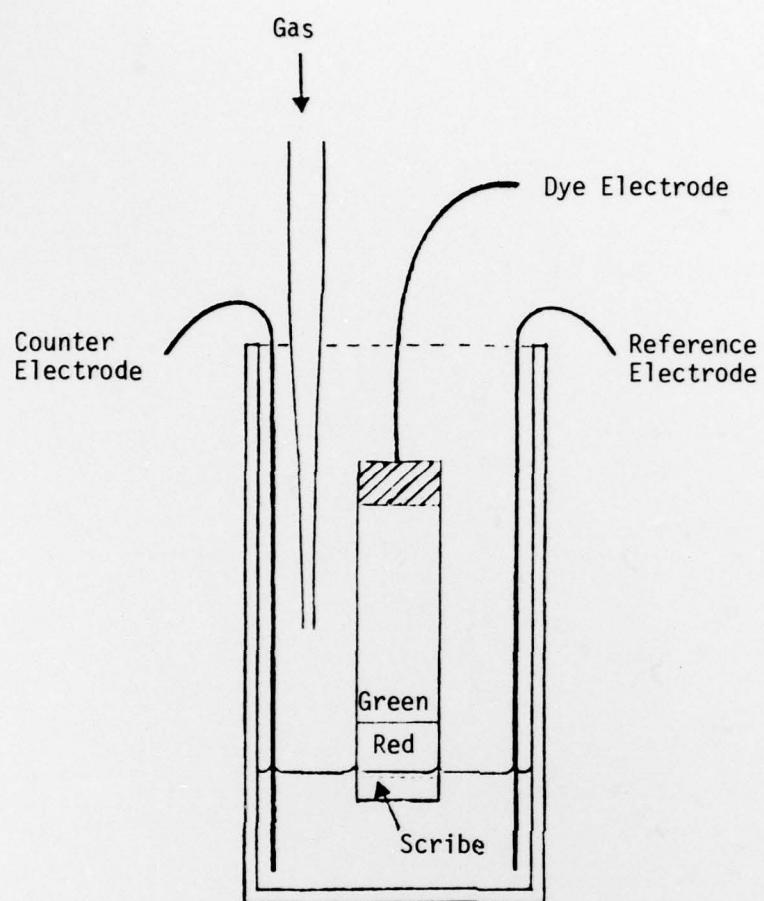


Fig. 1

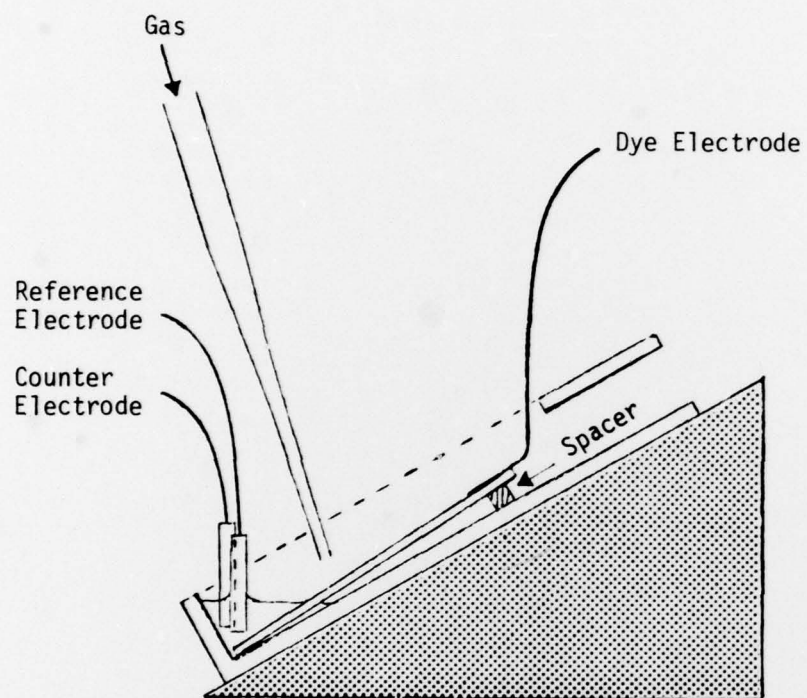


Fig. 2

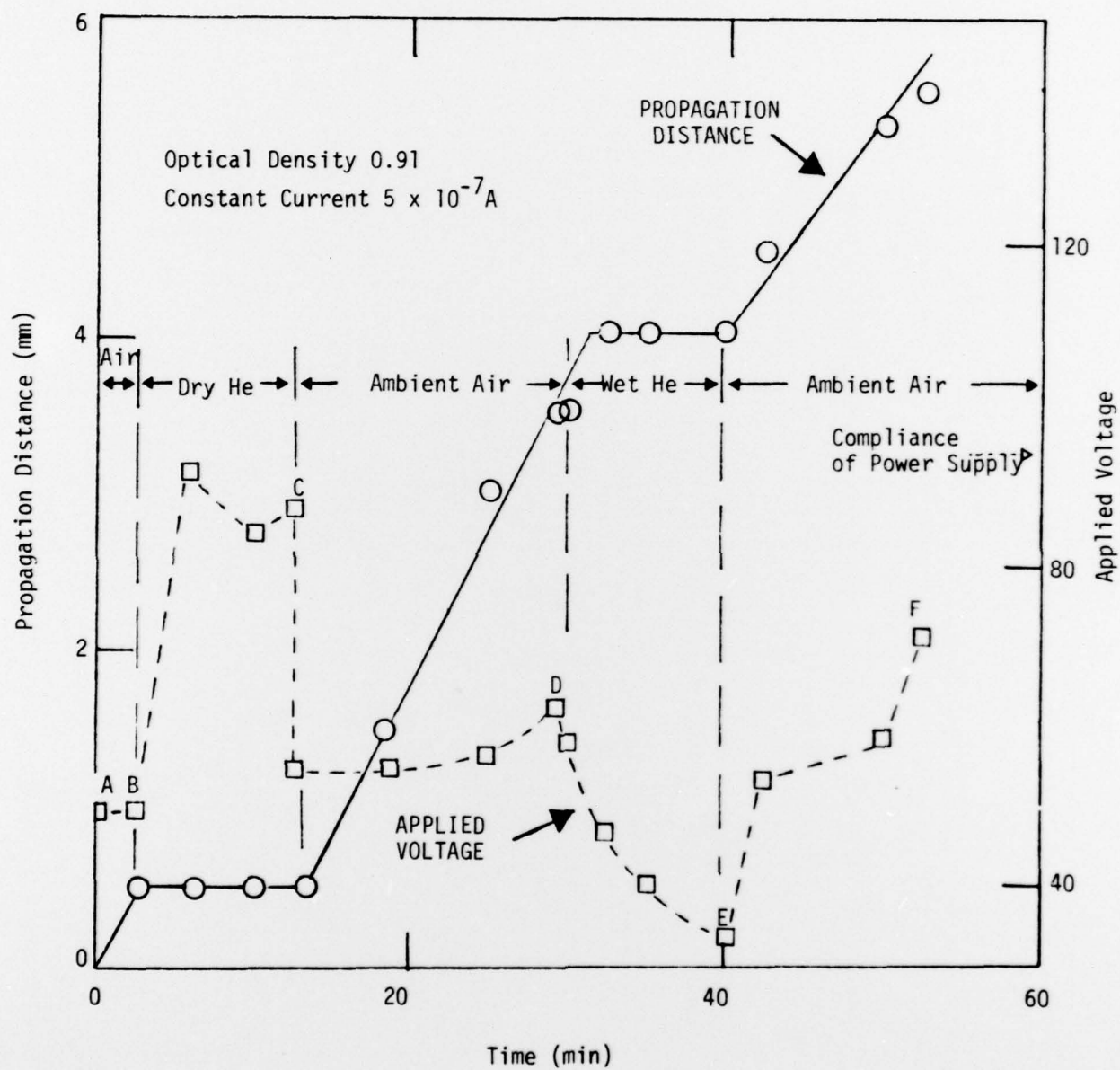


Fig. 3

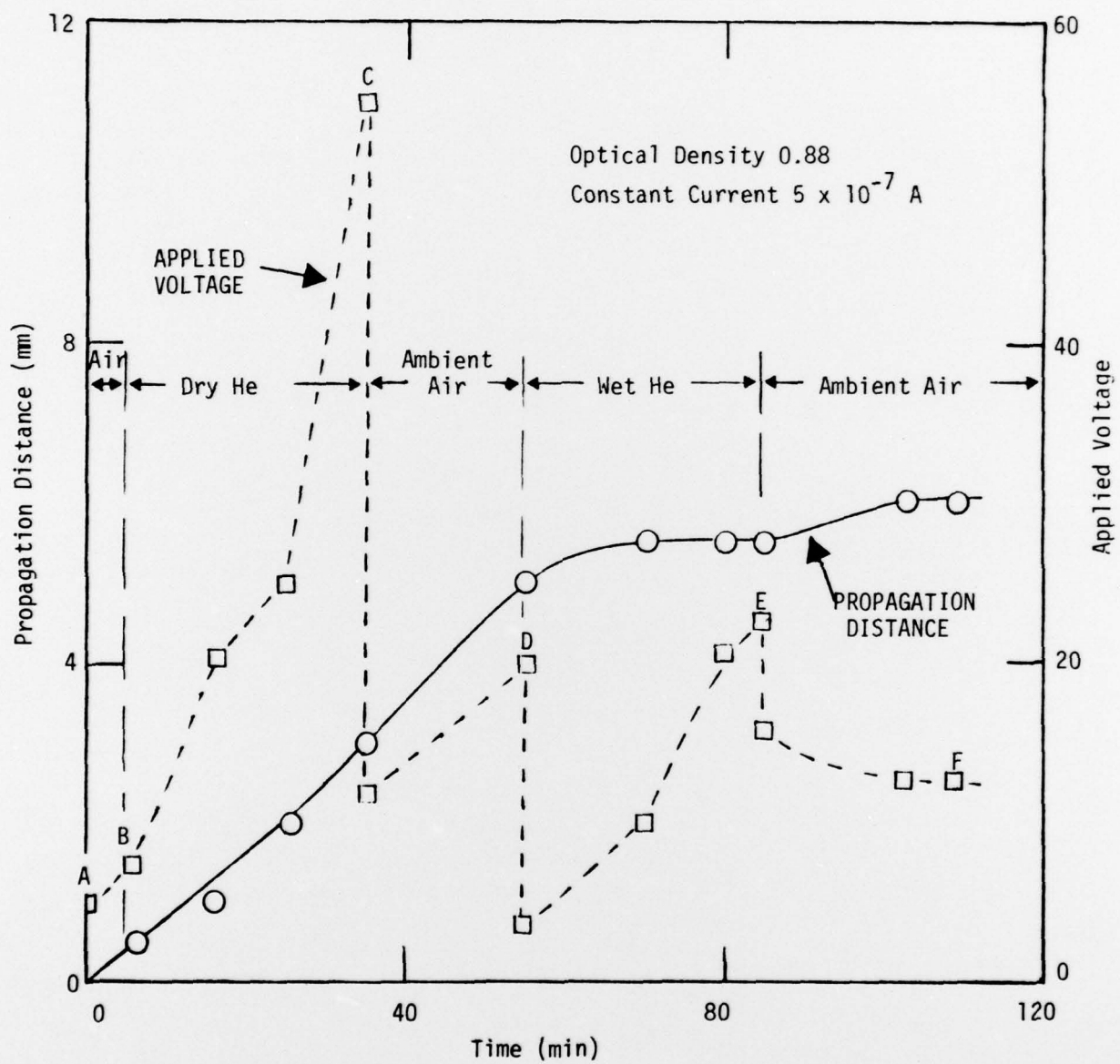


Fig. 4

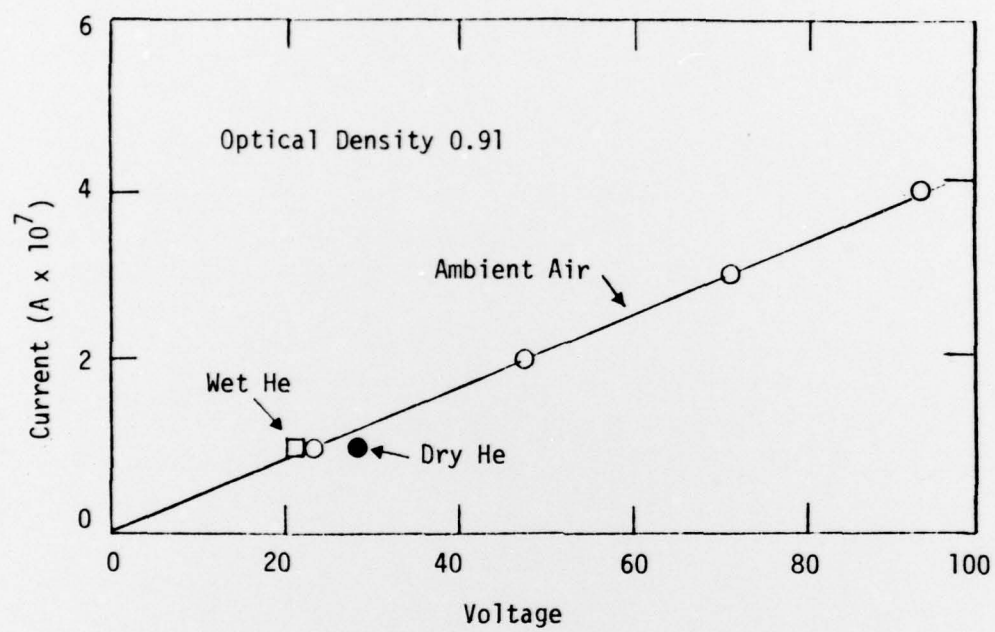


Fig. 5

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